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INFLUENCE OF MULTI-WALLED CARBON NANOTUBES ON THE THERMAL AND MECHANICAL BEHAVIOR OF CARBON/EPOXY COMPOSITES (PREPRINT)

Yuanxin Zhou, Farhana Pervin, Lance Lewis, and Shaik Jeelani

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Influence of Multi-walled Carbon Nanotubes on the Thermal and Mechanical Behavior of Carbon/Epoxy Composites

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ABSTRACT

In this study, a high-intensity ultrasonic liquid processor was used to obtain a homogeneous mixture of epoxy resin and multi-walled carbon nanotubes (CNTs). The CNTs were infused into epon 862 epoxy resin through sonic cavitation and then mixed with w curing agent using a high-speed mechanical agitator. The trapped air and reaction volatiles were removed from the mixture using a high vacuum. Flexural tests and fracture toughness tests were performed on unfilled and CNT-filled epoxy to identify the effect of adding CNTs on the mechanical properties of epoxy. The highest improvement in strength and fracture toughness was obtained with 0.3 wt% CNT loading. The nanophased matrix filled with 0.3 wt% CNT was then used with weave carbon fabric in a vacuum-assisted resin transfer molding (VARTM) set up to fabricate composite panels. Flexural tests, Thermogravimetric Analysis (TGA), and Dynamic Mechanical Analysis (DMA) were performed to evaluate the effectiveness of adding CNTs on the mechanical and thermal properties of the composite. The glass transition temperature, decomposition temperature, and flexural strengths were improved by infusing CNTs.

KEY WORDS: Thermal and mechanical properties, CNT, Carbon fiber reinforced epoxy

1. INTRODUCTION

Due to their high specific strength and specific stiffness, fiber-reinforced composites have become attractive structural materials not only in the weight-sensitive aerospace industry, but also in the marine, armor, automobile, railway, structural engineering, and sporting goods industries. Generally, the in-plane tensile properties of a fiber/polymer composite are defined by the fiber properties, while the compression properties and properties along the thickness dimension are defined by the characteristics of the matrix resin. Epoxy resin is the most commonly used polymer matrix for advanced composite materials. Over the years, many

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attempts have been made to modify epoxy by adding either rubber particles [1-2] or fillers [3-4] to improve the matrix-dominated composite properties. The addition of rubber particles improves the fracture toughness of epoxy, but decreases its modulus and strength. The addition of fillers, on the other hand, improves the modulus and strength of epoxy, but decreases its fracture toughness. The heat deflection temperature of epoxy is also improved by the addition of fillers [5]. In recent years, micro- and nano-scaled particles have been considered as filler material for epoxy to produce high performance composites with enhanced properties. For example, Wang et al. [6] used SiC whiskers along the interface of composite laminates during the lay-up process to improve their interlaminar fracture toughness. Sherman et al. [7] modified unidirectional carbon/epoxy by using very thin alumina platelets. Mahfuz and his co-workers [8] observed a 39% enhancement in flexural strength by infusing 1.5 wt% SiC nanoparticles in carbon/epoxy composite. Gojny et al. [9] reported the influence of carbon nanotubes on the mechanical and electrical properties of GFRP.

The primary objective of this paper is to determine the effect of adding carbon nanotubes (CNTs) on the mechanical properties of epoxy and carbon fabric/epoxy composites. Carbon nanotubes are excellent candidates for nano-reinforcing a variety of polymer matrices because of their strength, thermal conductivity, electrical capacity, stiffness, and thermal stability. Nanophased matrices based on polymers and carbon nanotubes have attracted great interest because they frequently include superior mechanical, electronic, and flame-retardant properties. Different polymer/CNT nanocomposites have been synthesized by incorporating CNTs into various polymer matrices, such as polyamides [10], polyimides [11-13], epoxy [14], polyurethane [15-16] and polypropylene [17-19]. These polymer-based nanocomposites derive their high properties at low filler volume fractions due to the high aspect ratio and high surface area to volume ratio of the nano-sized particles. According to Reynaud et al. [20], an interface of 1 nm thick represents roughly 0.3% of the total volume of the polymers in micro particle filled composites, whereas it can reach 30% of the total volume in nanocomposites. However, high specific surface areas cause a strong tendency to agglomerate, which reduces the strength of the nanocomposite as an effect of stress concentration. Optimal CNT loading in the matrix is a key parameter to developing a nanophased structure composite.

In this study, flexural and fracture tests were first performed on neat and CNT-filled epoxy to identify the optimal loading. After the optimal loading was determined, the CNT-filled epoxy matrix was used with weave carbon perform to fabricate composite panels. Thermogravimetric Analysis (TGA) and Dynamic Mechanical Analysis (DMA) were performed to evaluate their thermal performances. Flexural tests were performed to evaluate the mechanical performance of the resulting composite. Microscopic approaches were used to investigate the fracture behavior and mechanism of materials. Based on the experiment's results, a damage constitutive equation was developed to describe the stress-strain relationship of neat and nanophased carbon/epoxy composites.

2. MATERIALS

The epoxy used was Part A: Epon 862 (bisphenol F epoxy) and Part B: EpiCure curing agent W, both purchased from Miller-Stephenson Chemical Company, Inc. Carbon Nanotechnologies, Inc.

produced the multi-walled carbon nanotubes used in this study. The tube diameters range from 30 to 60 nm and the tube lengths range from 3 to $10 \mu m$. The weight fraction of the carbon nanotubes ranged from 0 wt% to 0.4 wt% to help identify the loading with the best mechanical properties. IM7 satin weave carbon fabric, $203 g/m^2$, 6k-sized fabric with a thickness of 0.23mm, manufactured by Hexcel INC, was used in this study.

3. OPTIMIZATION OF CNT/EPOXY RESIN

3.1 Manufacturing of nanophased matrix Figures 1A and 1B show the pictures of received carbon nanotubes at different magnifications. High specific surface area and cotton-like entanglements caused the formation of agglomerates [20]. Agglomerates of CNTs, called nanoropes, are difficult to separate and infiltrate with the matrix. For polymer matrix nanocomposites, high power dispersion methods, such as ultrasound and high-speed shearing, are the simplest and most convenient to improve the dispersion of nanosized fillers in a polymer matrix [13-14]. In this study, the components were mixed using a high-intensity ultrasonic processor.
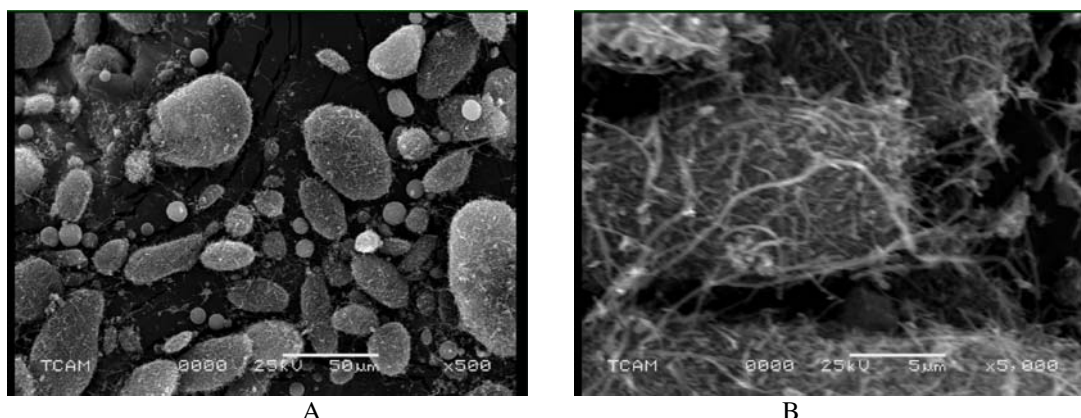

A   B

Figure 1 SEM pictures of as-received carbon nanotubes at the magnification
of 500 (A) and 5000 (B)

Pre-calculated amounts of carbon nanotubes and Epon 862 resin were carefully weighed and mixed together in a beaker. A high-intensity, ultrasonic irradiation mixed the CNTs and resin for an hour on pulse mode, 50 seconds on/25 seconds off (Ti-horn, 20 kHz Sonics Vibra Cell, Sonics & Materials, Inc). The beaker containing the mixture was submerged in an ice bath to keep it cool during the sonication process. Once the irradiation was complete, EpiCure curing agent W was added to the modified resin and mixed using a high-speed mechanical stirrer for about 10 minutes. The mix ratio of Epon 862 and W agent was 100:26. The mixing of epoxy and curing agent initially produced highly reactive, volatile vapor bubbles, which could create voids and detrimentally affect the properties of the final product. To reduce the chance of voids, the liquid was preheated to 80° C to reduce its viscosity, and a high vacuum system was used for about 30 minutes. After the bubbles were completely removed, the mixture was transferred to rectangular, Teflon-coated metal molds and cured for 4 hours at 120° C. The cured material was then trimmed. Finally, test samples were machined for thermal and mechanical characterization and all panels were post-cured at 170° C for 4 hours.

3.2 Flexural response

Table 1 Mechanical Properties of Neat and Nanophased Epoxy

	Modulus (GPa)	Strength (MPa)	Failure Strain (%)
Neat Epoxy	2.46	93.5	4.02
0.1% CNT	2.54	109	6.06
0.2% CNT	2.60	115	6.80
0.3% CNT	2.65	121	7.58
0.4% CNT	2.75	113	5.12

Flexural tests were performed according to ASTM D790-86 under a three-point bend configuration. The tests were conducted in a 10 KN servo-hydraulic testing machine equipped with a Test Ware data acquisition system. The machine was run under displacement control mode at a crosshead speed of 2.0 mm/min. All tests were performed at room temperature.
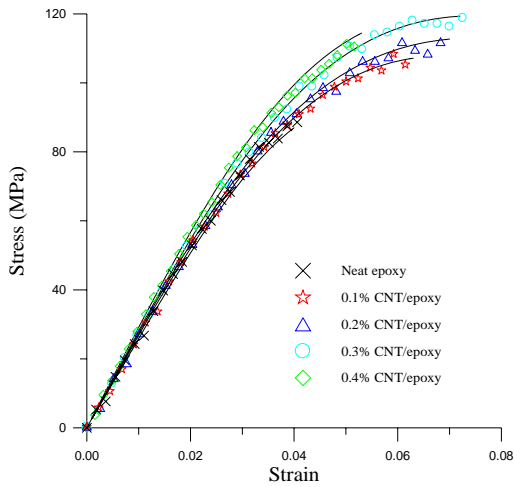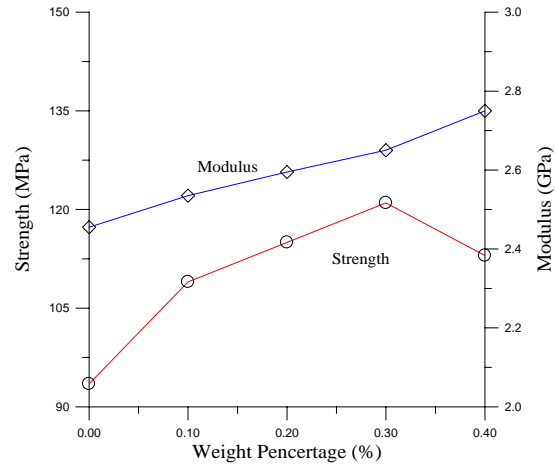

Figure 2 Stress-strain curves

Figure 3 Effect of CNT content on strength and modulus

Typical stress-strain behavior from the flexural tests is shown in Figure 2. All specimens failed immediately after the tensile stress reached the maximum value. The stress-strain curves showed considerable non-linearity before reaching the maximum stress, but no obvious yield point was found in the curves. Five specimens were tested for each condition; the average properties obtained from these tests are listed in Table 1.

The modulus of the nanophased epoxy increased continuously with higher CNT content (Table 1, Figure 2). The tensile modulus improved by 11.7% with an addition of 0.4 wt% of CNTs. However, the system with 0.3 wt% infusion is the best, with a 28.3% tensile strength enhancement (Table 1, Figure 2). The strength begins to degrade with 0.4 wt% loading, although the gain in modulus is maintained. See Figure 3 for the relationship between the modulus, strength, and CNT weight fraction. The dispersion of CNTs that restricts the mobility of polymer chains under loading improved the modulus and strength in small loadings. The high aspect ratio, high modulus, strength of CNTs, and good interfacial adhesion between the CNTs and matrix also contributed to the reinforcement. However, the decrease of strength with high CNT content

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can be attributed to the following two effects: First, the dispersion of CNTs is not uniform in higher loading systems. Acoustic cavitation is one of the most efficient ways to disperse nanoparticles with small loading into the pure materials. Previous results have also indicated that, using the acoustic cavitation method, the optimal loading of carbon nanofibers in epoxy is 2.0 wt% [21] and the optimal loading of SiC nanoparticles in epoxy is 1.0 wt% [22]. Second, voids may have decreased the strength. Choi et al. reported that few voids were produced during the fabrication process and that voids increased with higher nanoparticle contents [23].
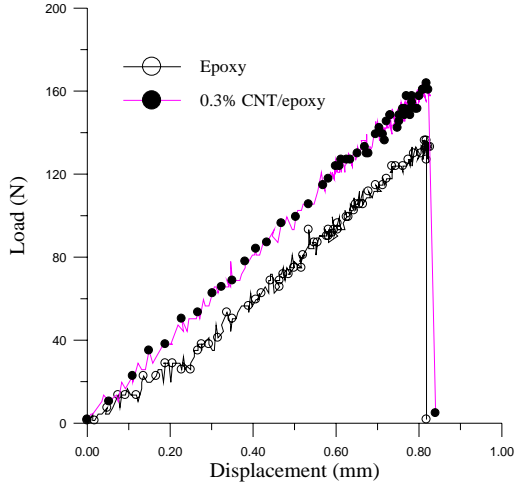
3.3 Fracture toughness
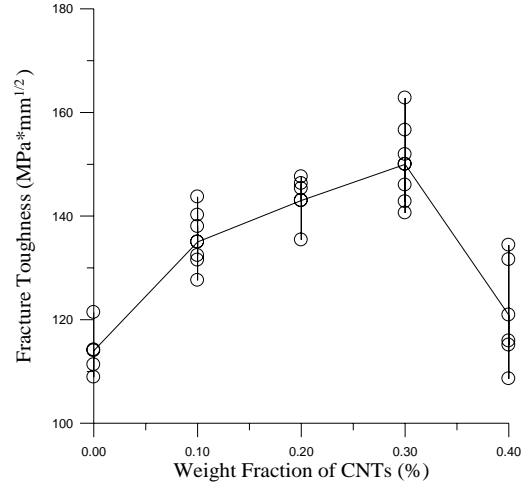

Figure 4 Load-displacement   Figure 5 fracture toughness vs. CNF content curve

Fracture toughness of neat and nanophased epoxy was determined from static three-point tests of the single edge notch specimens. Each of these specimens was cycled 100 times between 4% and 40% of the peak load at 1 Hz and then statically tested. During the static tests, the change in specimen length Δl was measured by recording the ram positions through the displacement transducer of the MTS machine.

The critical stress intensity factor, K_c, was calculated according to the following equation:

$$K_1 = \left(PS / Bw^{3/2} \right) f\left(a/w\right) \tag{1}$$

where P = applied load on the specimen
B = specimen thickness
w = specimen width
a = crack length
and

$$f\left(a/w\right) = \frac{3\sqrt{a/w}\left[1.99 - \left(a/w\right)\left(1 - a/w\right)\left(2.15 - 3.93\,a/w + 2.7\,a^2/w^2\right)\right]}{2\left(1 + 2\,a/w\right)\left(1 - a/w\right)^{3/2}}$$

Figure 4 shows the load-displacement diagrams of neat epoxy and of 0.3 wt% CNT/epoxy. Since non-linearity was seldom observed in load-displacement diagrams, the critical stress intensity

factor (K_{Ic}) of materials was calculated from the peak load of each load-displacement curve, and was plotted as a function of the CNT weight fraction (Figure 5). It shows that enhancement reaches a maximum for the critical stress intensity factor at 0.3 wt%. At the higher contents, fracture toughness decreased with filler loading.
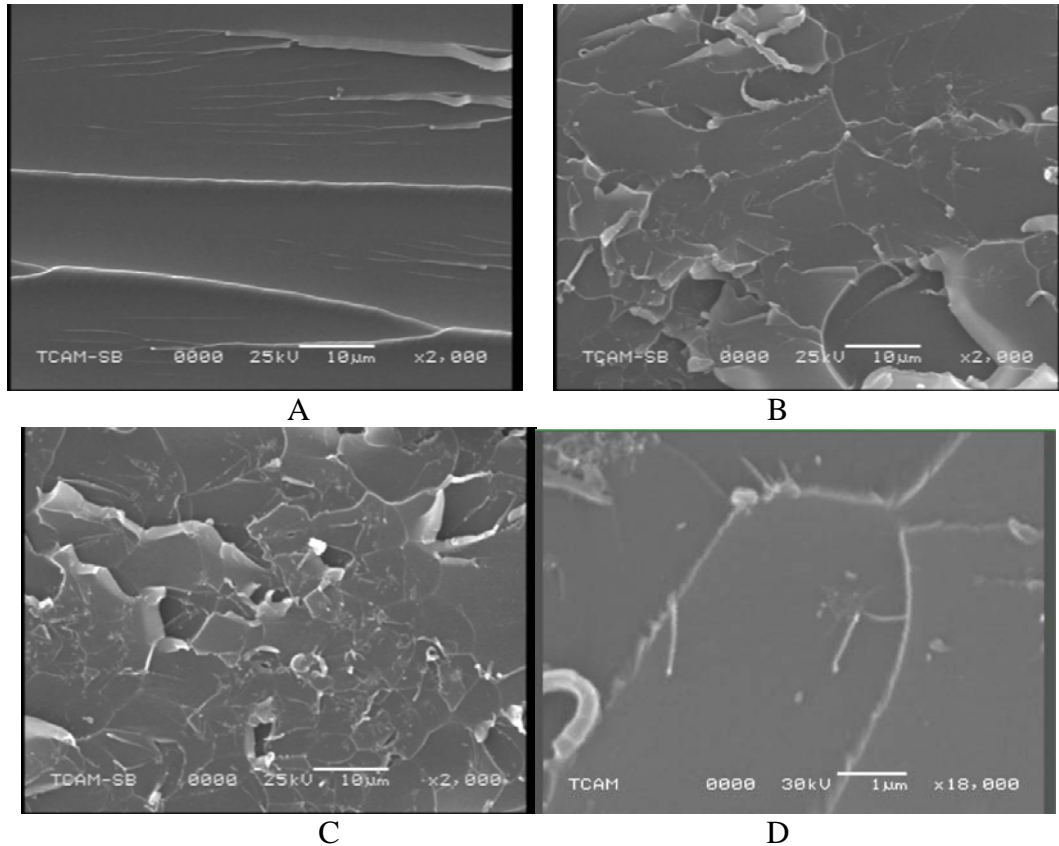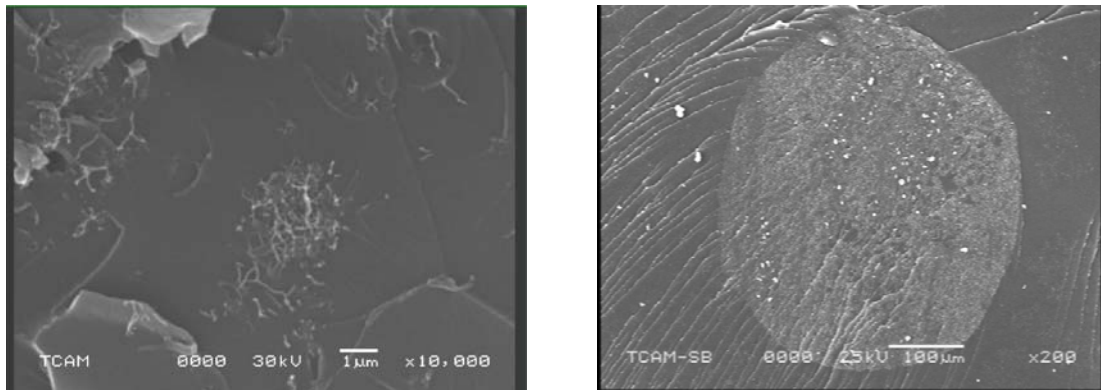
3.4 Fracture surface

Figure 6 Fracture surfaces of neat and nanophased epoxy (A: neat epoxy at magnification of 2000; B: 0.1%CNT/epoxy at magnification of 2000, C: 0.3%CNT/epoxy at magnification of 2000; D: 0.3%CNT/epoxy at magnification of 18000)

The fracture surfaces of the neat epoxy and its nanocomposites were comparatively examined using SEM. The initial crack occurred at the tension edge of both the neat and nanophased specimens. Neat epoxy resin exhibits a relatively smooth fracture surface and the SEM picture in Figure 6A indicates a typical fractography feature of brittle fracture behavior, thus accounting for the low fracture toughness of the unfilled epoxy. The distance between two cleavage steps is about 20 to 40 μm and the cleavage plane between them is flat and featureless. The fracture surfaces of the nanocomposites show considerably different fractographic features. For example, the failure surface of the nanocomposite containing CNTs is rougher, with the CNTs added into the epoxy matrix. Figures 6B and 6C show the fracture surfaces of 0.1 wt% CNT/epoxy and 0.3 wt% CNT/epoxy, respectively. The surface roughness increased with higher CNT content. Figures 6B (0.1 wt%) and 6C (0.3 wt%) also indicate that the size of the cleavage plane

decreased with higher CNT content. The high magnification pictures of the cleavage plane of the 0.3 wt% system (Figure 6D) show that CNTs were uniformly dispersed in the epoxy. The flat cleavage planes were formed by the network of cleavage steps and each plane contains at least one carbon nanotube. During the failure process, the crack propagation changed direction as it crossed CNTs. The bridge effect, which prevents crack opening, increased strength in the CNT/epoxy matrix. The SEM picture in Figure 6D shows that the size of the cleavage plane decreased to 5-10 μm after the infusion of the 0.3 wt% CNTs. The decreased cleavage plane and the increased surface roughness imply that the path of the crack tip is distorted because of the carbon nanotubes, making crack propagation more difficult.

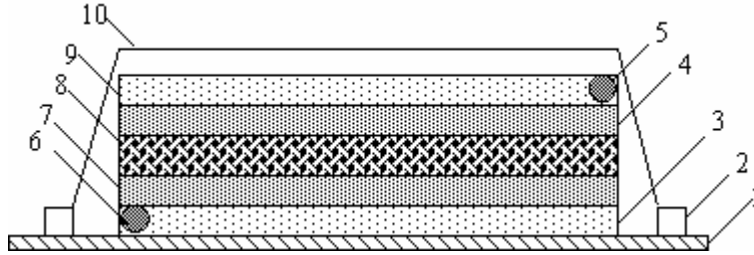A: Small agglomeration particle   B: Large agglomerated particle

Figure 7 Fracture surface of 0.4% CNT/epoxy

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When the CNT content increased to 0.4 wt%, small and large particle agglomerations of several carbon nanotubes were observed in the fracture surface (Figures 7A and 7B). At a low strain level, the agglomerated particle increased the stiffness of the material, but at a high strain level, the stress concentration caused by the agglomerated particle initiated a crack, which made the sample fail quickly. Therefore, a decrease in flexural strength and fracture toughness was observed in the 0.3 wt% CNT/epoxy.

4. CARBON-REINFORCED NANOPHASED EPOXY

4.1 Manufacturing of carbon/epoxy nanocomposite Based on the experimental results of the CNT-modified epoxy matrix, laminated composite panels were manufactured by using satin weave carbon fiber and 0.3 wt% CNT-modified epoxy. A schematic diagram of this manufacturing process is shown in Figure 8. The aluminum plate was laid on a flat surface and cleaned with acetone. A mold-releasing agent, free-coat, was added to the surface to allow easy release of the panel. Then, a layer of distribution mesh and a layer of porous Teflon were laid on the plate, respectively, for uniform resin distribution. Four layers of carbon fiber were used. Dry fabric preforms with required orientations were then laid out on top of the distribution mesh. Another layer of porous Teflon and distribution media were laid on the fibers. After stacking, the

complete assembly was covered with a heat-resistant vacuum bag, and infusion and suction lines were installed. Nanophased resin was infused from one end, and the other end was connected to a vacuum pump. The vacuum bagging is critical in composite manufacturing, and the cover was leakproof. Final test samples were machined for mechanical characterization. All panels were post-cured at 170°C for 4 hours. Using the same method, neat carbon/epoxy panels were fabricated to compare with the nanophased system.

1: Aluminum plate; 2: Boundary layer; 3: Distribution mesh; 4: Porous Teflon;
5: Inlet infusion tubing; 6: Exit extracts tubing; 7: Porous Teflon;
8: Carbon fiber perform; 9: Distribution mesh; 10 Vacuum bag
Figure 8 Fabrication of carbon/epoxy composite by using VARTM

4.2 Thermal stability and Tg Thermogravimetric analysis (TGA) was carried out to estimate the thermal stability and the amount of resin present in the neat and nanophased panels. The weight vs. temperature curve in Figure 9 indicates that the panel contains 19 wt% of epoxy resin and the rest is carbon fiber and nano-filler. In this study, we considered the derivative peaks as the decomposition temperature. As seen in Figure 10, the decomposition temperatures for neat carbon/epoxy and nanophased carbon/epoxy are 384.3°C and 387.7°C, respectively. CNTs have little effect on the thermal stability of carbon/epoxy composites. Figure 10 illustrates the DMA plots of the loss factor tanδ vs. temperature for neat and nanophased carbon/epoxy composites. Tg, determined from the peak position of tan δ, increased from 163.4^{o}C to 165.8^{o}C.
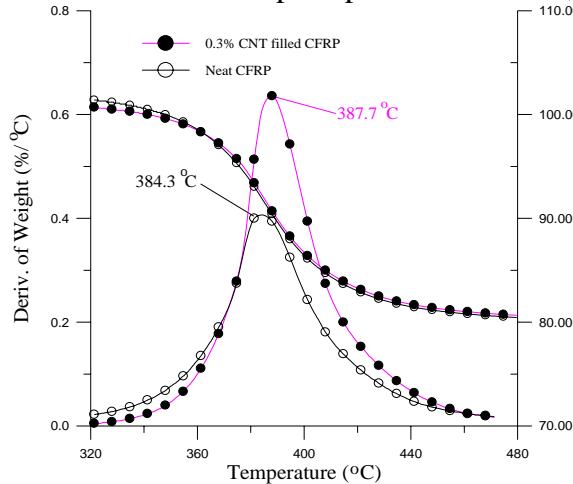

Figure 9 Thermogravimetric traces of neat and nanophased carbon/epoxy composite
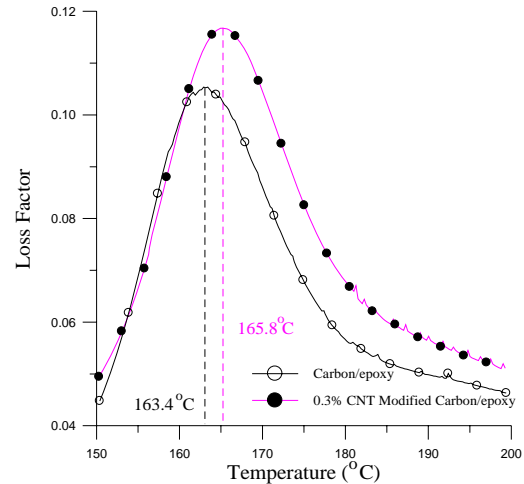
Figure 10 Loss factor vs. temperature curves of neat and nanophased carbon/epoxy composite

4.3 Mechanical properties Flexural tests were performed on an MTS servohydraulic test machine equipped with a 10KN load cell. Test coupons were prepared according to ASTM D790 (three-point loading system). Five specimens with a span-to-depth ratio of 16:1 were cut from both neat and nanophased laminate. Typical specimen dimensions were 56mm in length, 25mm in width, and 2mm in thickness. Span length was kept at 32.2mm. Displacement controlled, three-point bending tests were carried out at a crosshead speed of 0.9mm/min until specimens failed. *TestWare-SX* PC-installed software on the machine was used to control the movements and record loads and corresponding displacements. Two parameters were evaluated from stress-strain curves: modulus (E) and failure strength $\left(\sigma_b\right)$.
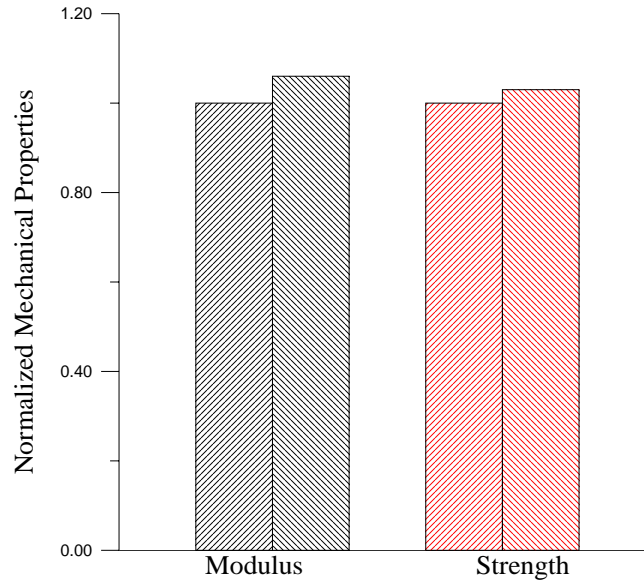

Figure 11 Experimental stress-strain curves

Mechanical properties of neat and nanophased laminates are compared in Figure 11. The nanophased system showed an approximately 3% increase in flexural strength and a 6% increase in modulus, compared to the neat system. There are two possible reasons for the higher mechanical properties of the carbon fabric-reinforced laminate infused with CNTs. First, the increased strength of the epoxy matrix caused by the CNTs, as observed in the first part of this study, can be regarded as the reason for the strength increase. The second reason is that CNTs increase the crack propagation resistance by the bridging effect, which improves interlaminar strength [24].

4.3 Fracture surface To understand the gain in the mechanical properties of the nanophased composite, the fracture surface of the neat and nanophased composites were investigated by using SEM. Figure 12A shows the low magnification fracture micrographics of the nanophased carbon/epoxy. The neat system has the same results. Fiber breakage, interface debonding, and layer delamination all can be observed in Figure 12A. However, at high magnification, SEM pictures show the different results. For neat carbon/epoxy (as shown in Figure 12B), the surface of the fiber was clean, and no matrix adhered to the fiber. The fracture surface of the matrix was

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flat, and some cracks were seen in the matrix side near the fiber/matrix interface. These results all indicated that the fabric and matrix were poorly connected and that interfacial bonding was weak.
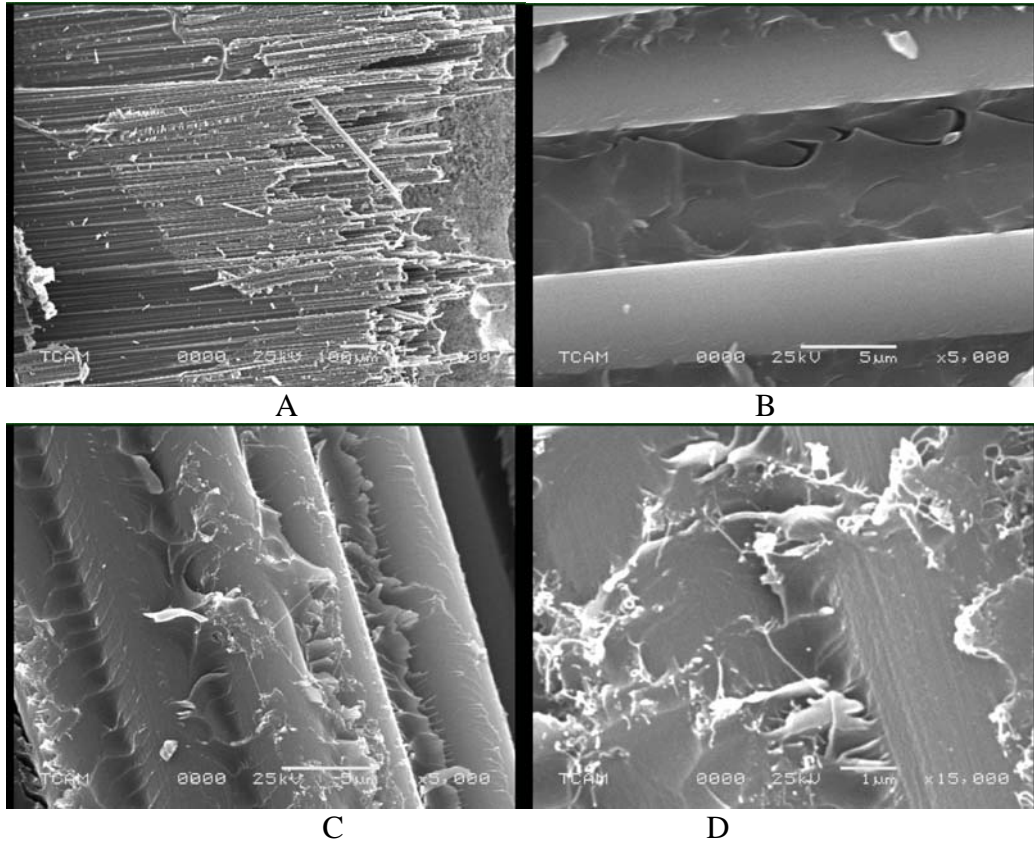

Figure 12 Fracture surface of carbon/epoxy composite
(A: at low magnification; B: neat carbon/epoxy at magnification 5000; C: nanophased carbon/epoxy at magnification 5000; D: re-agglomerated CNTs in composite)

Contrary to the above, the fracture surface of the nanophased composite in Figure 12C shows that a thin film of matrix material adhered to the fibers, indicating that the interface was strong. The surface of the matrix was rougher than the neat composite, and CNTs were randomly distributed in the matrix. In this case, the strengthened matrix held the carbon fabrics together. Strong fiber/matrix interface is another reason that explains the strengthening behavior of the nanocomposite. Figure 12D shows the fracture of nanophased carbon/epoxy at a magnification of 15,000. The dispersion of CNTs in the carbon/epoxy was not uniform, and some agglomerated CNTs were observed. It is caused by re-agglomeration during degassing of VARTM. According to Gojny's results [9], degassing and injection in the VATRM process leads to the formation of a nanotube/epoxy suspension and facilitates the re-agglomeration of the nano-fillers, as a result of the increasing surface area. In order to suppress the re-agglomeration, it is necessary to stabilize the nanotube suspension or reduce surface area of nano-fillers.

5. Conclusions

Mechanical and thermal tests were conducted on CNT-modified epoxy and carbon/epoxy composites. Based on the experiment's results, the following conclusions are reached:

1. Carbon nanotubes were infused in epoxy by the ultrasonic cavitation method, by different loading. 0.3 wt% CNT loading in epoxy resin showed the highest improvement in tensile strength, as compared to neat and other nanophased systems.

2. 0.3 wt% CNT-modified epoxy was used to fabricate carbon/epoxy nanocomposites. Improvements in flexural strength, glass transition temperature, and decomposing temperature were observed.

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